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मानक

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“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 5296 (1995): Chloroform, Pure and Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



“ज्ञान से एक नये भारत का निर्माण”

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“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS 5296 : 1995

(Reaffirmed 2005)

Edition 3.2

(2002-05)

भारतीय मानक

क्लोरोफार्म, शुद्ध तथा तकनीकी — विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

**CHLOROFORM, PURE AND TECHNICAL —
SPECIFICATION**

(Second Revision)

(Incorporating Amendment Nos. 1 & 2)

ICS 71.080.15

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BUREAU OF INDIAN STANDARDS

MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG

NEW DELHI 110002

Price Group 7

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1969 and revised in 1979 to incorporate the limits for moisture content and impurities like methyl chloride, methylene chloride and carbon tetrachloride along with their methods of test. Also the requirements for relative density and distillation range have been suitably modified with respect to the type of stabilizer used.

The committee responsible for the preparation of this standard decided to revise it again in the light of the latest development and the quality of the material presently being made available. In the present revision of the standard, the requirement of distillation range has been updated, details of test methods have been suitably modified and requirement for colour has been introduced.

Chloroform, also sometimes known as trichloromethane, is used as a solvent for waxes, fats, oils, rubber, resins, etc. Besides being a good solvent, it is used as a cleansing agent, in fire extinguishers to lower the freezing temperature of carbon tetrachloride and for the manufacture of refrigerants of the chloro-fluoro methane series. It is also used as an analytical reagent, again as solvent and eluent. The requirements of chloroform for medical purposes are laid down in the Indian Pharmacopoeia.

The anaesthetic properties of chloroform are well known, though due to its toxic effect, this use is being abandoned. On inhalation, it irritates the mucous membrane; prolonged inhalation will bring on paralysis which is often fatal (*see* 5 and 6.3).

This edition 3.2 incorporates Amendment No. 1 (January 1999) and Amendment No. 2 (May 2002). Side bar indicates modification of the text as the result of incorporation of the amendments.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CHLOROFORM, PURE AND TECHNICAL — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for chloroform, pure and technical.

1.1.1 It does not cover pharmaceutical grade of the material.

2 NORMATIVE REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard. The standard contains provisions which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent revisions of the standards indicated below:

IS No.	Title
265 : 1987	Hydrochloric acid (<i>third revision</i>)
266 : 1977	Sulphuric acid (<i>second revision</i>)
878 : 1975	Graduated measuring cylinders (<i>first revision</i>)
915 : 1975	One-mark volumetric flasks (<i>first revision</i>)
1070 : 1992	Water for general laboratory use (<i>third revision</i>)
1575 : 1990	Separating funnels (<i>first revision</i>)
2303 : 1963	Method of grading glass for alkalinity
2362 : 1993	Determination of water by the Karl Fischer method (<i>first revision</i>)
2618 : 1989	Test tube (<i>first revision</i>)
2837 (Part 2) : 1977	Porcelain crucibles and basins: Part 2 Basins (<i>first revision</i>)
4161 : 1967	Nessler cylinders
4905 : 1968	Methods for random sampling

3 GRADES

3.1 There shall be two grades of the material, namely:

- a) *Pure* — Generally used for the manufacture of refrigerants of the chloro-fluoro methane series and as an analytical reagent; and
- b) *Technical* — Suitable for other industrial uses.

4 REQUIREMENTS

4.1 Description

The material shall be highly refractive, non-flammable, heavy, very volatile, sweet-testing liquid with characteristic odour. It shall be miscible with alcohol, benzene, ether, carbon tetrachloride and carbon disulphide.

4.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods referred in col 5 of Table 1.

4.2.1 Specific Requirement

For the alcohol stabilized product, the requirements for relative density, distillation range and moisture content shall be as follows:

- i) Relative density at 27°C/27°C 1.466 to 1.474
- ii) Distillation range, temperature (being corrected to a pressure of 760 mm Hg) The difference between the temperatures (running points at which 2 and 97 percent of volume taken have been collected shall not exceed 2°C and the range shall include the temperature of 61.3°C).
- iii) Moisture content, percent by mass, *Max* 0.05

Table 1 Requirements for Chloroform, Pure and Technical

(Clause 4.2)

Sl No.	Characteristics	Requirements		Method of Test, Ref to Annex (5)
		Pure (3)	Technical (4)	
i)	Relative density at 27°/27°C	¹⁾ 1 477 to 1 483	¹⁾ 1 474 to 1 484	A
ii)	Distillation range, the temperature being corrected to a pressure of 760 mm Hg	The difference between the temperature (running points) at which 2 and 97 percent of the volume taken have been collected shall not exceed 1°C and the range shall include the temperature of 61 3°C	The difference between the (running points) at which 2 and 97 percent of the volume taken have been collected shall not exceed 2°C and the range shall include the temperature of 61 3°C	B
iii)	Residue on evaporation, percent by mass, <i>Max</i>	0 002	0 01	C
iv)	Foreign organic matter	To pass the test	—	D
v)	Free chlorine	To pass the test	To pass the test	E
vi)	Acidity, percent by mass (as HCl), <i>Max</i>	Neutra ¹	0 02	F
vii)	Aldehydes, percent by mass, <i>Max</i>	0 000 5	—	G
viii)	Ketones, percent by mass, <i>Max</i>	0 005	—	G
ix)	Phosgene	To pass the test	—	H
x)	Chloride, percent by mass, <i>Max</i>	0 000 1	—	J
xi)	Moisture content, parts per million, <i>Max</i>	100	—	K
xii)	Methyl chloride, methylene chloride and carbon tetrachloride, percent by mass, <i>Max</i>	0 025	—	L
xiii)	Colour in Hazen units, <i>Max</i>	15	15	M

NOTE — The above requirements are for the hydrocarbon stabilized product

¹⁾ The correction factor within the range of 25 to 35°C is +0 0018 for every degree celsius fall and -0 0018 for every degree Celsius rise in temperature

4.3 A suitable stabilizer as agreed to between the supplier and the purchaser shall be added to the material. The maximum quantities of stabilizers recommended are as follows:

- a) Hydrocarbon stabilized material 35 ± 15 ppm
- b) Alcohol stabilized material 1.5 percent (v/v)

5 PRECAUTION IN HANDLING

On inhalation or ingestion, it leads to narcosis, preceded by a stage of excitation, then follows loss of reflexes and consciousness. Hence, it should be handled carefully. If a person shows symptoms of chloroform poisoning, he should be immediately removed to fresh air, suitable first aid should be given and physician should be called.

6 PACKING, STORING AND MARKING

6.1 Packing

The material shall be packed as agreed to

between the supplier and the purchaser. The packing generally used is described below:

“Pure grade of material used for analytical purposes shall be packed in amber-coloured glass bottles, with well-fitting stoppers and non-reacting lining (gaskets).

The material stabilized with hydrocarbon may be packed in galvanized iron drums with non reacting gaskets on the stoppers. The drums shall be free from contaminations like oil and grease, rust, etc, and shall be tested previously at a pressure of 1 bar. The material stabilized with ethyl alcohol may be packed in tin cans, tinned mild steel drums or stainless steel drums which shall be dry and free from contaminants like oil and grease, rust, etc. The filling holes shall be hermetically sealed with suitable non-reacting gaskets. The latter two shall be previously tested at a pressure of 0.5 bar.”

6.2 Storing

The material of both pure and technical grade

shall be stored in tightly closed containers, indoors, in cool places and shall be protected from sun rays and moisture.

6.3 Marking

The containers shall be labelled with the following information:

- a) Manufacturer's name and recognized trade-mark, if any;
- b) Grade of the material;
- c) Net mass;
- d) Batch number; and
- e) The following cautionary markings in clear bold letters:

CHLOROFORM

Warning ! Vapour harmful.

Use only with adequate ventilation.

Avoid breathing vapour.

Avoid prolonged or repeated contact with the skin.

Do not take internally.

6.3.1 The pure grade used for analytical purposes may also be labelled with the complete analytical data for the characteristics given in Table 1.

6.3.2 BIS Certification Marking

The containers may be marked with the Standard Mark.

6.3.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers and producers may be obtained from the Bureau of Indian Standards.

7 SAMPLING

Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Annex N.

8 QUALITY OF REAGENTS

8.1 Unless specified, otherwise, pure chemicals and distilled water (see 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[Table 1, SI No. (i)]

DETERMINATION OF RELATIVE DENSITY

A-1 OUTLINE OF THE METHOD

In this method, mass of equal volumes of the material and water at the same temperature are compared using relative density bottle.

A-2 APPARATUS

A-2.1 Relative Density Bottle — 25 ml capacity.

A-2.2 Water Bath — maintained at $27.0 \pm 0.2^\circ\text{C}$.

A-2.3 Thermometer — Any convenient thermometer of a suitable range with 0.1 or 0.22 deg sub-divisions.

A-3 PROCEDURE

A-3.1 Clean and dry the relative density bottle, weigh and then fill with recently boiled and cooled water at 27°C . Fill to overflowing by holding the relative density bottle on its side in such a manner as to prevent entrapment of air bubbles. Insert the stopper and immerse in the

water-bath. Keep the entire bulb of the bottle completely covered with water and hold at that temperature for 30 minutes. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, cool and weigh. Calculate the mass of water. Again clean and dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

A-3.2 Calculation

$$\text{Relative density at } 27^\circ\text{C}/27^\circ\text{C} = \frac{A - B}{C - B}$$

where

A = mass in g of relative density bottle filled with the material;

B = mass in g of the clean and dry relative density bottle; and

C = mass in g of the relative density bottle filled with water.

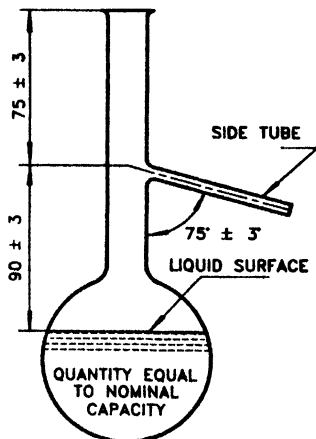
ANNEX B

[Table 1, Sl No. (ii)]

DETERMINATION OF DISTILLATION YIELD

B-1 APPARATUS

B-1.1 Distillation Flask — of the shape and dimensions shown in Fig. 1.



All dimensions in millimetres.

FIG. 1 DISTILLATION FLASK

B-1.2 Thermometer — of the mercury in glass partial, immersion type, having a range between 0 to 100°C, graduated at each 0.2 deg and having a maximum error of ± 0.4 deg. The thermometer shall, have an overall length of 385 mm, maximum and stem diameter 5.5 to 8.0 mm.

B-1.3 Liebig Condenser — made of Type 1 glass (graded according to IS 2303 : 1963) with a wall thickness of 1.0 to 1.5 mm and conforming to the shape and dimensions given in Fig. 2.

B-1.4 Receiver — 100 ml capacity, with dimensions and graduations as shown in Fig. 3.

B-1.5 Rectangular Draught Screen — made of 0.8 mm thick metal sheet with the dimensions shown in Fig. 4 and open at the top and bottom. It shall comply with the following requirements:

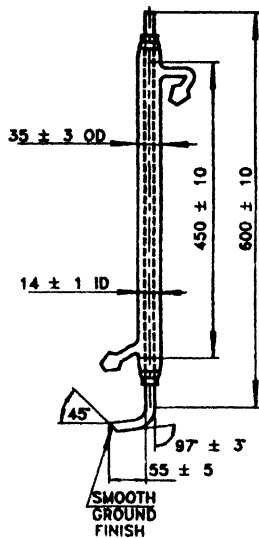
- a) In each of the two narrower sides of the draught screen, there shall be two circular air vents 25 mm in diameter, situated below the asbestos shelf, as shown in

Fig. 4. In each of the four sides of the draught screen there shall be three air vents with their center 25 mm above the base of the draught screen. These holes shall occupy the positions shown in Fig. 4, the diameter of each of the holes centrally situated in the wider sides being 25 mm, and of the remaining ten holes 12.5 mm. At the middle of each of the wider sides, there shall be a vertical slot for the side-tube of the distillation flask, with dimensions as shown in Fig. 4 and cut downwards from the top of the screen. A removable shutter conforming to the dimensions shown in Fig. 5 shall be provided for closing vertical slot not in use.

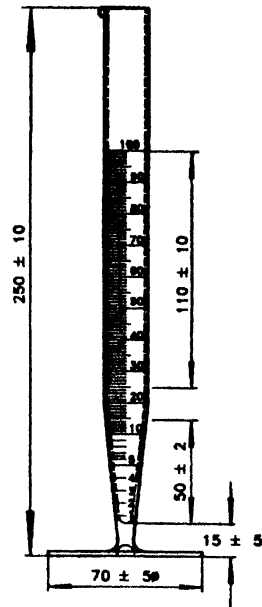
- b) A shelf of hard asbestos board, 6 mm in thickness, and having a central hole 110 mm in diameter, shall be supported horizontally, in the screen and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for asbestos shelf may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.
- c) In one of the narrower sides of the screen a door shall be provided having dimensions and position as shown in Fig. 4. In each of the narrower sides of the screen a mica window shall be placed centrally with the bottom of the window on a level with the top of the asbestos shelf. The dimensions and position of the windows are shown in Fig. 4.
- d) An asbestos board 150 × 150 × 6 mm in size having a central hole 50 mm in diameter shall be so placed on the asbestos shelf that the two holes are approximately concentric and the distillation flask when in position completely closes the hole of the asbestos board.

B-1.6 Electric Heater, Gas Burner or Other Flame Type Heater

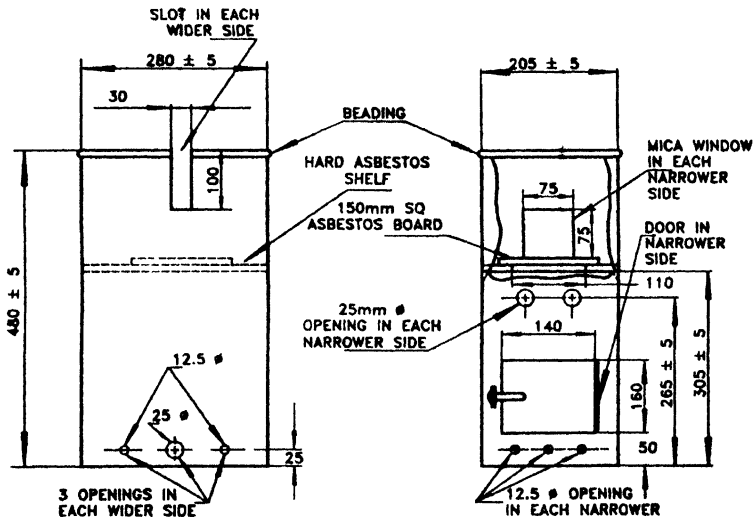
Any suitable heater or burner that enables the distillation to be carried out as described in B-3.



All dimensions in millimetres
FIG. 2 LIEBIG CONDENSER



All dimensions in millimetres
FIG. 3 RECEIVER



All dimensions in millimetres.

FIG. 4 RECTANGULAR DRAUGHT SCREEN

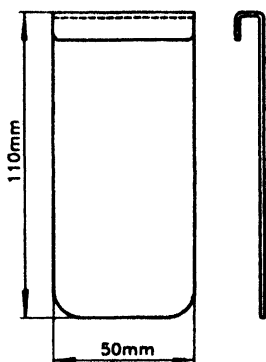


FIG. 5 REMOVABLE SHUTTER

B-2 ASSEMBLY OF THE APPARATUS

B-2.1 Assemble the apparatus as shown in Fig. 6.

B-2.1.1 Position of Thermometer

The thermometer shall be held concentrically in the neck of the flask by means of a well-fitting stopper of a material which is not

attacked by the liquid, and the junction of capillary tube and the main bulb of the thermometer shall be maintained in level with the lower edge of the joint of the side-tube and the neck of the flask. The stopper shall project about 10 mm above the top of the neck of the flask. When the thermometer is fixed in position as indicated above, the immersion line on the thermometer shall be in the neighbourhood of the top of the cork holding the thermometer.

B-2.1.2 Support for Flask

The asbestos board [see B-1.5(d)] shall be so placed on the top of the asbestos shelf of the draught screen that the two holes are approximately concentric. The flask shall then be placed in position and pressed down so as to close completely the hole in the asbestos board.

B-2.1.3 Connection of Flask to Liebig Condenser

The flask is so connected to the condenser that the end of the side-tube projects at least 25 mm into the condenser and is coaxial with it.

B-3 PROCEDURE

Measure 100 ml of the material in the receiver. Transfer the liquid as completely as possible to the distillation flask and add a few small pieces of clean, dry porous earthenware or stoneware. Place the flask, thermometers and receiver in position and ensure that the condenser has a steady supply of water. Apply heat at uniform rate so regulated that the first drop of distillate falls from the end of the condenser in 5 to

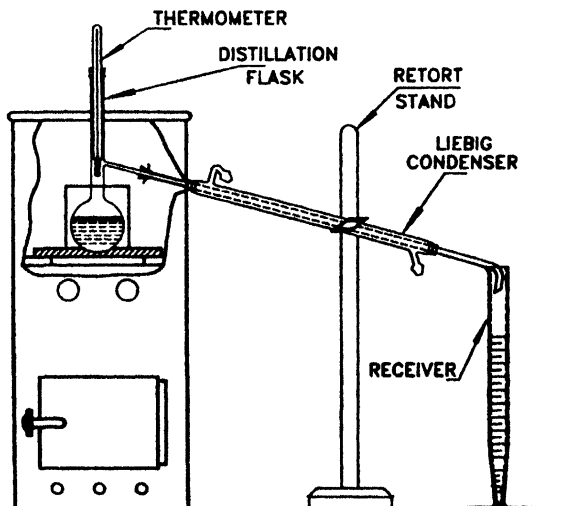


FIG. 6 ASSEMBLY OF APPARATUS

10 minutes. Further regulate heat so that the distillate is collected at the rate of 3 to 4 ml per minute. Note the temperature in the thermometer when 2 ml and 97 ml of the distillate has collected in the receiver. Discard any liquid on the side of the flask.

B-4 CORRECTION OF THERMOMETER READING

B-4.1 Error of Scale

In all thermometer readings, make the corrections as indicated on the certificate of the instrument.

B-4.2 Correction for Barometric Pressure

If the barometric pressure prevailing during

the determination is 760 mm Hg, no correction need be applied to the observed temperature of the boiling range. If the barometric pressure deviates from 760 Hg, the observed temperature shall be corrected as follows:

- a) For every 10 mm above 760 mm Hg, subtract 0.40 deg from the observed temperature of boiling range to get the specified temperature range as per Table 1; and
- b) For every 10 mm below 760 mm Hg, add 0.40 deg to the observed temperature of boiling range to get the specified temperature range as per Table 1.

NOTE - These corrections are valid only for pressure above 700 mm Hg

ANNEX C

[Table 1, Sl No. (iii)]

DETERMINATION OF RESIDUE ON EVAPORATION

C-1 APPARATUS

C-1.1 Porcelain Basin — 150 ml capacity, conforming to IS 2837 (Part 11) : 1977.

C-1.2 Water Bath

C-1.3 Oven — capable of maintaining temperature between 105°C to 110°C.

C-2 PROCEDURE

Clean the basin and dry in a desiccator. Weigh it to the nearest 0.0002 g. Take 100 ml of the material in the basin. Evaporate the material over a water bath in a fume cupboard to almost

dryness. Further heat in the oven at 105°C to 110°C for 30 minutes. Cool in a desiccator and weigh accurately. Repeat the heating for 15 minutes, followed by cooling and weighing till, two consecutive weighings do not differ by more than 0.5 mg.

C-3 CALCULATION

Residue on evaporation percent by mass = $\frac{M}{d}$

where

M = mass in g of the residue; and

d = density of the material (see Annex A).

ANNEX D

[Table 1, Sl No. (iv)]

TEST FOR FOREIGN ORGANIC MATTER

D-1 APPARATUS

D-1.1 Glass Cylinder — about 2 cm in diameter.

D-2 REAGENTS

D-2.1 Concentrated Sulphuric Acid — See IS 266 : 1977.

D-2.2 Formalin — containing not less than 37 percent by mass of formaldehyde.

D-2.3 Potassium Dichromate

D-2.4 Cobalt Chloride

D-2.5 Concentrated Hydrochloric Acid — conforming to IS 265 : 1987.

D-2.6 Standard Solution for Matching Colours

Prepare by mixing 50 ml each of the following two solutions:

- a) **Solution A** — Dissolve 0.1 g of potassium dichromate weighed to the nearest 0.0002 g in water and make up the volume to 500 ml.

IS 5296 : 1995

b) *Solution B* — Dissolve 2.975 g of cobalt chloride in water, add 1.5 ml of concentrated hydrochloric acid and make the volume to 500 ml with water.

D-3 PROCEDURE

D-3.1 Take 15 ml of the material in the glass cylinder and add 15 ml of concentrated

sulphuric acid. Shake the mixture carefully and add 4 drops of formalin. Keep aside for 10 minutes. Then match the colour of the solution with 30 ml of the standard solution (see **D-2.6**) taken to a similar glass cylinder.

D-3.2 The material shall be taken to have passed the test, if no brown colour darker than that of the standard solution appears.

ANNEX E

[Table 1, *Sl No.* (v)]

TEST FOR FREE CHLORINE

E-1 OUTLINE OF THE METHOD

The material is shaken with 3,3'-dimethylnaphthidine solution and the colour developed, if any, is noted.

E-2 APPARATUS

E-2.1 Graduated Measuring Cylinder — 50 ml, glass stoppered (see IS 878 : 1975).

E-3 REAGENT

E-3.1 3, 3'-Dimethylnaphthidine Solution

Dissolve 0.01 g of finely ground 3, 3' dimethy-

lnaphthidine in 5 ml of glacial acetic acid and dilute rapidly with water to 200 ml. Store the solution in the dark.

E-4 PROCEDURE

E-4.1 To 50 ml of the material contained in the graduated measuring cylinder, add 5 ml of the 3, 3'-dimethylnaphthidine solution and shake the cylinder for 30 seconds and keep in the dark.

E-4.2 The material shall be considered as showing no free chlorine if no pink colour is developed within one minute

ANNEX F

[Table 1, *Sl No.* (vi)]

DETERMINATION OF ACIDITY (AS HYDROCHLORIC ACID)

F-1 REAGENTS

F-1.1 Bromothymol Blue Indicator

Dissolve 0.5 g of water soluble bromothymol blue crystals in 100 ml of water. Dilute 80 ml of this solution to 500 ml with methyl alcohol.

F-1.2 Standard Sodium Hydroxide Solution — 0.01 N.

F-2 PROCEDURE

To 100 ml of water, neutral to bromothymol blue, add 100 ml of the material and shake vigorously for one minute at least. Add bromothymol blue indicator and titrate with standard sodium hydroxide solution until a bluish green end point persists for 1 minute.

F-3 CALCULATION

$$\text{Acidity (as HCl), percent by mass} = \frac{3.65 \times V \times N}{v \times d}$$

where

V = volume in ml of standard sodium hydroxide solution;

N = normality of standard hydroxide solution;

v = volume in ml of the material taken for the test; and

d = relative density of the material.

ANNEX G[Table 1, *Sl No.* (vii) and (viii)]**DETERMINATION OF ALDEHYDES AND KETONES****G-1 GENERAL**

This test is applicable only to pure grade of the material.

G-2 APPARATUS

G-2.1 Separating Funnel — 25 ml capacity.

G-3 REAGENTS

G-3.1 Mercuric Chloride — solid.

G-3.2 Potassium Iodide — solid.

G-3.3 Sodium Hydroxide Solution — 9 N.

G-3.4 Nessler's Reagent

Dissolve 8 g of mercuric chloride in 50 ml of water and add a solution of 7.4 g potassium iodide in 50 ml of water. Decant red precipitate of mercuric iodide and wash three times

with water. Add 5 g of potassium iodide and just that quantity of water which would dissolve the precipitate completely. Make up the solution to 100 ml, with sodium hydroxide solution. Decant to free from any sediment and keep in well-stoppered bottle in a dark place.

G-4 PROCEDURE

G-4.1 Take 3 ml of the material in the separating funnel, add 10 ml of ammonia-free water and shake for 5 minutes. Take 5 ml of the aqueous phase and add to it 40 ml of water and 5 ml of Nessler's reagent.

G-4.2 The limits for aldehydes ketone specified in Table 1 shall be taken not to have been exceeded if no turbidity or precipitate is produced within one minute.

ANNEX H[Table 1, *Sl No.* (ix)]**TEST FOR PHOSGENE****H-1 GENERAL**

This test is applicable only to pure grade of the material.

H-2 APPARATUS

H-2.1 Test Tube — not more than 10 mm in outer diameter (see IS 2618 : 1989).

H-2.2 Pipette**H-3 REAGENT**

H-3.1 Barium Hydroxide Solution — saturated. Filter before use.

H-4 PROCEDURE

H-4.1 Take 10 ml of the material in the test-tube. Add carefully by means of pipette without disturbing the surface of the liquid, sufficient quantity of saturated barium hydroxide solution to form a layer about 5 mm deep. Observe the junction of liquids in strong diffused light.

H-4.2 The material shall be taken to have passed the test for phosgene, if no white film is seen at the liquid junction.

ANNEX J

[Table 1, Sl No. (x)]

DETERMINATION OF CHLORIDES**J-1 GENERAL**

This test is applicable only to pure grade of the material.

J-2 APPARATUS

J-2.1 Separating Funnel — 100 ml capacity
(see IS 1575 : 1990).

J-3 REAGENT**J-3.1 Silver Nitrate Solution**

Dissolve 17 g of silver nitrate (AgNO_3) in water and make up the volume to 1 000 ml.

Store in a cool place away from light in blue bottles.

J-4 PROCEDURE

J-4.1 Take 17 ml of the material in a separating funnel and add 25 ml of water. Shake well for 5 minutes. Allow the liquids to separate and discard the chloroform layer. Take 10 ml of the aqueous phase in a test tube and add 5 drops of silver nitrate solution. Shake and watch for any turbidity.

J-4.2 The limit for chlorides prescribed in Table 1 shall be taken not to have been exceeded if no turbidity is produced within one minute.

ANNEX K

[Table 1, Sl No. (xi)]

DETERMINATION OF MOISTURE**K-1 PROCEDURE**

Take 100 ml of the hydrocarbon stabilized or

50 ml of alcohol stabilized material (see 4.3) and determine the moisture content as prescribed in IS 2362 : 1973.

ANNEX L

[Table 1, Sl No. (xii)]

DETERMINATION OF METHYL CHLORIDE, METHYLENE CHLORIDE AND CARBON TETRACHLORIDE**L-1 OUTLINE OF THE METHOD**

The chlorinated compounds like methyl chloride, methylene chloride and carbon tetrachloride present in chloroform in small amounts are determined by gas chromatography.

L-2 APPARATUS**L-2.1 Gas Chromatograph**

Any commercially available gas chromatograph dual column with a thermal conductivity detector may be used. The operating conditions of one of the suitable columns are given below:

<i>Column</i>	
Material	Stainless steel or copper
Length	3.6 m
Internal diameter	4 mm, approximately
External diameter	6 mm, approximately
Support	Chromosorb W (60 to 80 mesh)
Stationary phase	Di-2 ethyl hexyl sebacate
Preparation of the stationary phase	20 percent by mass of Di-2 ethyl hexyl sebacate on the chromosorb W (60 to 80 mesh)

L-2.2 Detector — thermal conductivity cell type.

L-2.3 Recorder — full scale deflection : 1 second.

L-2.4 Microlitre Syringe — 50 μ l capacity.

L-3 TEST SUBSTANCES

L-3.1 The following components are estimated:

- a) Methyl chloride;
- b) Methylene chloride; and
- c) Carbon tetrachloride.

L-3.2 Synthetic Standard

Mix 0.01 percent each of methyl chloride, methylene chloride and carbon tetrachloride in pure chloroform.

L-4 PROCEDURE

L-4.1 Operating parameters of gas chromatograph with the above column (L-2.1) are as follows:

- a) Injection port temperature 140°C
- b) Column temperature 80 \pm 1°C
- c) Carrier gas Hydrogen (99.99 percent purity minimum on v/v basis). Flow rate: 80 ml/minute, inlet pressure: approximately 0.6 bar
- d) Detector Temperature 265 \pm 1°C
- e) Bridge current 200 mA
- f) Recorder Chart speed 12.5 mm/min.

L-4.2 Calibration

Internal normalization is the method used for calibration of the results of the analysis. Because of low contents of impurities it is not

necessary to use correction factors for converting the percentage of the areas into percentage by mass, so that these factors in the calculation formula have the value $K = 1$. In case of examples with higher contents of impurities, there is a need for determining correction factors; this should be done by using a synthetic standard solution. The mixture is run through the chromatograph. This would give the positions at which each component would elute. By comparison of the actual values obtained for the standard and the composition of the standard prepared the correction factor for the component may be calculated.

L-4.3 Sample Injection

Rinse the microlitre syringe twice with actone and dry it for at least 2 minutes. Take 20 μ l of the synthetic standard in the syringe and inject it into the gas chromatograph. Put the instrument on attenuation and obtain a standard chart. Repeat the injection using 20 μ l of the sample under test using the same injection technique as in the preparation of the standard chart. It is imperative that the same injection technique is used and the operation is not used with any other type of solvent.

L-4.4 Interpretation of Chromatogram

Elution order of the components is methyl chloride, methylene chloride, carbon tetrachloride and chloroform. Identification is done with the aid of a test mixture.

L-5 CALCULATION

$$X_i = \frac{100 \times K_i \times A_i}{T_a}$$

where

- X_i = percentage by mass of component i,
 K_i = correction factor of component,
 A_i = peak area due to component i [peak height (X) width at half height], and
 T_a = sum of total peak area of all the components (calculated as correction factor of each individual component \times peak height \times width at half height).

ANNEX M

[Table 1, Sl No. (xiii)]

TEST FOR COLOUR

M-1 OUTLINE OF THE METHOD

The colour of the material is compared with that of the colour standard and expressed in terms of Hazen colour units. The Hazen colour is defined as the colour of an aqueous solution, containing 1 part per million of platinum in the form of the chloroplatinic acid and 2 part per million of cobaltous chloride.

M-2 APPARATUS

M-2.1 Nessler Cylinders — two, 100 ml capacity (see IS 4161 : 1967).

M-2.2 One Mark Volumetric Flasks — 250 ml and 500 ml capacity (see IS 915 : 1975).

M-3 REAGENTS

M-3.1 Cobaltous Chloride Hexahydrate — solid.

M-3.2 Hydrochloric Acid — conforming to IS 265 : 1987.

M-3.3 Chloroplatinic Acid

Dissolve 250 mg of platinum in small quantity of aquaregia contained in a glass of porcelain basin by heating on a water bath. When the metal dissolved, evaporate the solution to

dryness. Add 1 ml of hydrochloric acid and again evaporate to dryness. Repeat this operation twice.

M-4 PREPARATION OF COLOUR STANDARD

M-4.1 Dissolve 0.50 g of the cobaltous chloride hexahydrate and whole of the chloroplatinic acid in 50 ml of the hydrochloric acid. Warm, if necessary, to obtain a clear solution and after cooling, pour into the 500-ml one-mark graduated flask. Dilute with water to the mark.

M-4.2 Pipette 7.5 ml of the solution (see M-4.1) into a 250-ml one-mark graduated flask and dilute with water to the mark. The diluted solution corresponds to a colour of 15 Hazen units and should always be freshly prepared.

M-5 PROCEDURE

M-5.1 Fill one of the Nessler cylinders to the mark with the material to be tested, and the other with the colour standard. Compare the colour using a white background.

M-5.2 The material shall be taken to have passed the test if the colour of the sample is not darker than that of the colour standard.

ANNEX N

(Clause 7)

SAMPLING OF CHLOROFORM, PURE AND TECHNICAL

N-1 GENERAL REQUIREMENT OF SAMPLING

N-1.1 The sampling instrument shall be clean and dry.

N-1.2 Precautions shall be taken to protect the sample, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

N-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by shaking or stirring or both by suitable means, or by rolling.

N-1.4 The samples shall be placed in suitable, clean dry, and airtight, metal, or dark or amber glass containers on which the material has no action.

N-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

N-1.6 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date sampling, and the month and year of manufacture of the material.

N-1.7 Samples shall be stored in the dark.

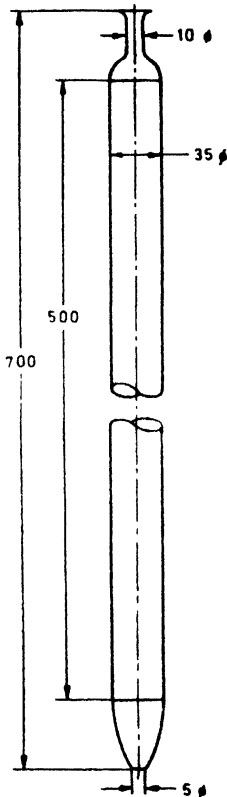
N-2 SAMPLING INSTRUMENT

N-2.1 The following sampling instrument may be used:

- a) Sampling bottle or can, for taking samples from tanks or drums;
- b) Sampling tube, for taking samples from bottles or small containers.

N-2.1.1 Sampling Tube

Made of metal or thick glass, 35 mm in diameter and 700 mm in length (see Fig. 7). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end.



All dimensions in millimetres.

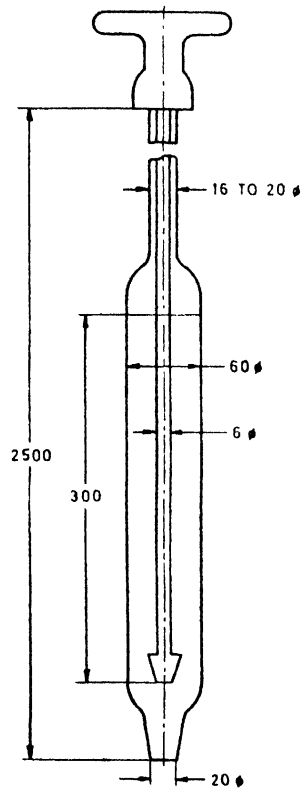
FIG. 7 PIPETTE

N-2.1.2 Sampler with Ground Closure

Made of steel and having a capacity of 500 to 1 000 ml (see Fig. 8).

N-2.1.3 Sampling Bottle or Can

Consists of a weighed glass or metal containers with removable stopper or top to which is attached a light chain (see Fig. 9). The bottle



All dimensions in millimetres.

FIG. 8 STEEL SAMPLER WITH GROUND CLOSURE

or the can is fastened to a suitable pole. For taking a sample, the bottle or the can is lowered into the tank to the required depth and the stopper is then removed by means of the chain.

N-2.1.4 Continuous Sampler

It shall be made of steel (see Fig. 10).

N-3 SCALE OF SAMPLING**N-3.1 For Tanks and Drums**

Each tank or drum shall be sampled separately.

N-3.2 For Bottles and Small Containers

Each lot (see N-3.2.2) shall be sampled separately.

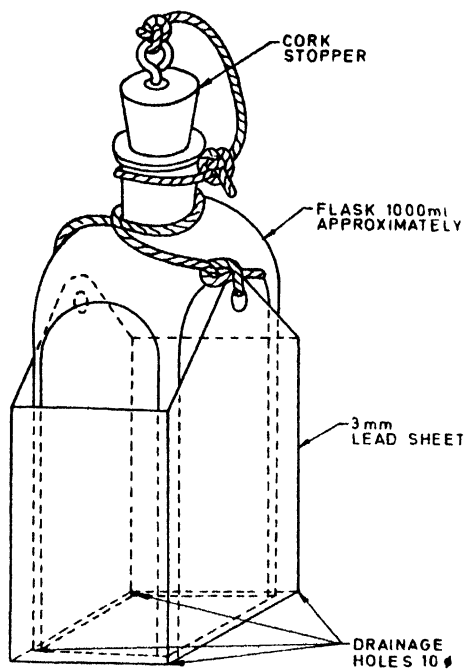


FIG. 9 BOTTLE

N-3.2.1 Lot

In any consignment, all the containers of the same grade and drawn from a single batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different grades, the containers belonging to the same batch and grade shall be grouped together and each such group shall constitute a separate lot.

N-3.2.2 The number of containers (n) to be selected from a lot shall depend on the size of the lot (N) and shall be in accordance with Table 2.

N-3.2.3 These containers shall be selected from the lot at random. In order to ensure randomness of selection, a random number table may be used. For guidance and use of random number table, IS 4905 : 1968 may be referred. In the absence of random number table, the following procedure may be adopted:

Starting from any container in the lot, count the containers as 1,2,3,.....etc, up to r and so on, in one order. Every r th container thus counted shall be withdrawn to

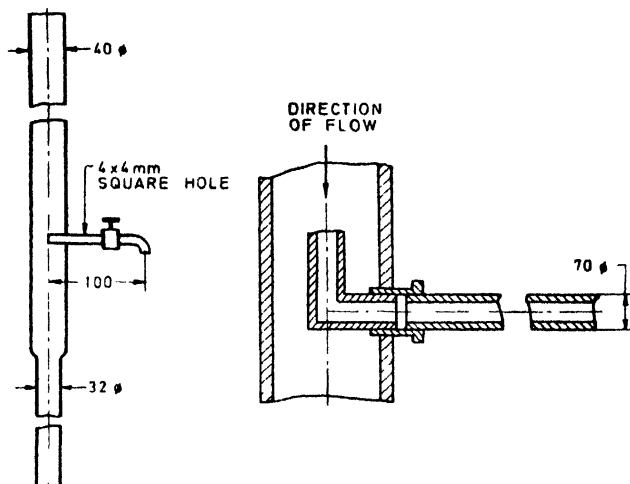
constitute the sample where r is the integral part of N/n (N and n being the lot size and sample size respectively).

N-4 TEST SAMPLE AND REFEREE SAMPLE

N.4.1 From Tanks and Drums

Table 2 Scale of Sampling
(Clause N-3.2.2)

Lot Size (N)	No. of Containers to be Selected (n)
(1)	(2)
Up to 4	All
5 „ 10	5
11 „ 20	6
21 „ 30	7
31 „ 50	8
51 „ 75	9
76 „ 100	10
101 „ 125	11
126 „ 150	12
151 „ 200	13
201 and above	14



All dimensions in millimetres

FIG. 10 CONTINUOUS SAMPLER

As far as possible, samples from a tank or drum should be drawn during the operation of filling. In that case, equal amounts of the material shall be collected at regular intervals so as to get a total amount of about 1 800 ml. Where it is not possible to take a sample during filling, the material shall be drawn from different position and depths with the sampling bottle or can after thoroughly agitating the material so as to ensure a fair amount of homogeneity. The total amount of the material collected shall be thoroughly mixed and divided into three equal portions, one for the purchaser, another for the supplier and the third for the referee.

N-4.2 From Bottles and Small Containers

From each of the bottles or containers selected according to N-3.2.3, a small representative portion of the material shall be drawn with the help of the sampling tube. Equal quantities of the material so drawn from the various containers shall be thoroughly mixed to form a test sample of about 1 800 ml. This shall be

divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

N-4.3 All the test samples shall be transferred to separate sample containers and sealed and labelled with full identification particulars. The referee test sample bearing the seal of both the purchaser and the supplier shall be kept at a place agreed to between the two and shall be used in case of a dispute.

N-5 TESTS

N-5.1 Tests for the determination of all the requirements given in this specification shall be performed on the composite sample obtained in N-4.1 or N-4.2.

N-6 CRITERIA FOR CONFORMITY

N-6.1 The lot shall be declared as conforming to this specification if all the test results satisfy the prescribed requirements.

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 323 01 31, 323 33 75, 323 94 02

Telegrams: Manaksanstha
(Common to all offices)

Regional Offices:

	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	{ 323 76 17 323 38 41
Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Kankurgachi KOLKATA 700054	{ 337 84 99, 337 85 61 337 86 26, 337 91 20
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022	{ 60 38 43 60 20 25
Southern : C. I. T. Campus, IV Cross Road, CHENNAI 600113	{ 235 02 16, 235 04 42 235 15 19, 235 23 15
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